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Technical Report #7

THE BENDING OF MOLYBDENUM SINGLE CRYSTALS

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Contract Nona 248(05)

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Several investigations have been conducted on pure bending of face-centered cubic metals.⁽¹⁾⁽²⁾⁽³⁾ Structural changes as a result of "bend-gliding," and the importance of bending and constraints during conditions of axial stressing, have been noted.⁽⁴⁾⁽⁵⁾ It was evident that the mechanism by which the bending deformation occurs is more complex than the case of simple shear. Studies of the bending of body-centered cubic metals from the aspect of the crystallographic mechanism appear to be lacking. Molybdenum single crystals were therefore subjected to deformation by bending in order to determine the reaction of the body-centered cubic lattice to bending stresses.

It might be expected for the bending of single crystals that lattice rotation as represented by the axis of compression would proceed toward the pole of the active slip plane and that rotation of the tensile stress axis should indicate the slip direction.⁽¹⁾ It was therefore believed that a study of the tension and compression sides of bent molybdenum single crystals should give data concerning the operative slip system. Specific problems which are pertinent to the over-all picture of plastic deformation were also investigated, e.g., whether the constraints offered by pure

* Research Associate, Metallurgy Section, The Johns Hopkins University, Baltimore 18, Maryland.

** Associate Professor of Metallurgy, The Johns Hopkins University.

*** Assistant Professor of Metallurgy, The Johns Hopkins University.

(1) References will be found at the end of the paper.

bending give rise to the deformation band, and also the nature of the asterism occurring on the tension and compression sides during the stages of deformation.

EXPERIMENTAL PROCEDURE

Eight single crystal specimens were grown from sintered molybdenum rods 1/8 inch diameter using the method described in a previous publication.⁽⁶⁾ The purity of this material was reported as 99.9 pct. as described elsewhere.⁽⁷⁾ The specimens were 1/8 to 1/10 inch in diameter x 7 inches long, with single crystals approximately 1 to 2 inches in length occupying the entire center sections of each specimen. The specimens were electrolytically polished using an electrolyte of 300 cc methyl alcohol, 60 cc H_2SO_4 , 130 cc HCl, and a current density of 4 amperes per square inch.

Each specimen was then loaded in a bending apparatus similar to that used by Yen and Hibbard,⁽¹⁾ as shown in Figure 1. The load was applied by means of a spring and screw arrangement through four ball-bearing surfaces set 1.5 inches apart. Either the inner two bearings or the outer two (a, b, Figure 1) were attached to a movable steel plate (d) through which the load was transmitted. Since the apparatus was mounted on a track of the X-ray apparatus, the tension or compression side of the specimen could be set up for exposure by interchanging the bearings. For example, when the inner two bearings were attached to the movable plate, the specimen was set up for exposure on the tension side, and when the outer two bearings were attached to the movable plate the specimen was exposed to the X-rays on the compression side.

The load was measured by a calibrated clip gage (e in Figure 1), using SR-4 type A-1 strain gages; the deflection was measured similarly using a calibrated clip gage (f). Load and deflection were measured by employing strain indicators. The load could be measured to a minimum of

0.0005 pound and the deflection to 0.0001 inch.

X-ray back-reflection Laue photographs of the same position on each specimen were taken before bending, and at successive stages during the bending with the load applied but not increasing. The lattice rotations were then plotted in a standard stereographic projection from the photograph. The entire course of deformation was followed in detail on the compression side for specimens M-35, M-83, M-88, M-90 and M-181, and on the tension side for specimens M-85, M-89 and M-184. Considerable accuracy could be attained in the determination of the above lattice rotations with the present experimental arrangement, since it was only necessary to keep the film to specimen distance constant without any re-alignment of the specimen. However, determination of the lattice rotations in the opposite sides of the above specimens could not be obtained with the same accuracy, particularly where the total rotation was small, since only the initial and final orientations were determined and re-alignment of the specimen was required after unloading. The nature of the asterism occurring on the tension and compression sides of the bent specimens was noted from the X-ray photographs.

The eight specimens, M-35, M-83, M-85, M-88, M-89, M-90, M-181 and M-184 were bent through the following final angles: 13° , 10° , 16° , 10° , 16° , 11° , 12° , and 15° respectively. Micrographic examination was carried out to determine the characteristics of the deformation markings appearing on the surface of the electropolished specimens after bending, and also to determine the pole of the slip plane. Measurements on those specimens where the slip lines could be observed satisfactorily were made at each 15° for several positions around the specimen axis on the metallograph. An indicator was attached to the specimen along a reference mark that was also used for the X-ray determination of the orientation. The angle

of rotation was measured on a protractor attached to the metallograph stage as noted by the indicator. The angle between the specimen axis and the slip marking was then determined at a magnification of $\times 400$. The orientation of the specimens after deformation was always used in correlating slip traces measured on the metallograph with the orientation determined from x-ray.

RESULTS AND DISCUSSION

Lattice Rotation

The specimen axis orientations for the initially unstrained crystals are shown in Figure 2 in a standard cubic stereographic projection. The specimen axis or lattice rotations which occurred on the compression side during the bending tests are given in Figures 3 and 4. It is evident from Figure 3 that specimens M-35 and M-89 showed a definite rotation toward the $(\bar{1}01)$ pole although the evidence for specimen M-88 is only slight. In Figure 4 the specimen axis rotation for specimens M-83 and M-181 was again clearly toward the $(\bar{1}01)$ pole. However, some scatter in the rotations was obtained for specimen M-90, although there is an apparent rotation toward the $(\bar{1}01)$ pole.

The specimen axis shift for the tension side of specimens M-85, M-89 and M-184 after increased amounts of bending are shown in Figure 5. The rotations were clearly toward the $[1\bar{1}1]$ direction for each of these three specimens. It should be noted that the $[1\bar{1}1]$ direction is contained in the $(\bar{1}01)$ plane.

The results presented in Figures 3 and 4 have shown that the lattice rotations on the compression side of bent single crystals of molybdenum were always toward a $\{110\}$ pole. Also, rotations on the tension side, as illustrated in Figure 5, were toward the $[1\bar{1}1]$ direction. It is well understood that for a single crystal loaded in compression, the lattice

rotation takes place in such a manner that the slip plane approaches the compression plane; for tensile loading the slip direction approaches the stress or tension axis.⁽⁸⁾ It would also be expected from the bending work of Yen and Hibbard⁽¹⁾ that rotation of the lattice on the compression side would proceed toward the pole of the active slip plane and that rotation on the tension side should indicate the direction of glide. The present results, therefore, appear to indicate that the slip planes are of the type $\{110\}$ and the slip direction $\langle 111 \rangle$ for molybdenum single crystals deformed at room temperature. These results for lattice rotation in bending also support earlier findings of Chen and Maddin⁽⁷⁾ in the plastic behavior of molybdenum single crystals. It is also worthy of note that the change of orientation on the compression and tension sides of a molybdenum single crystal (e.g., M-89), appears to resemble that of two separate crystals deforming under compression and tension loading respectively. Yen and Hibbard⁽¹⁾ also reached a similar conclusion for the transverse bending of single crystals of aluminum.

Asterism

X-ray back-reflection patterns, in conjunction with the load-deflection tests, revealed that the distortion of the Laue spots appeared when the specimen was subjected to loading beyond the yield. A progressive increase in the amount of asterism on the tension or compression sides was also noted with increasing plastic deformation due to bending (Figure 6). The extent of asterism was usually somewhat larger on the tension side than on the compression side, although about equal amounts of asterism were obtained on the tension and compression sides of M-35. X-ray photographs of the neutral region, i.e., 90° from the tension or compression sides where the stress should be theoretically zero, indicated very little

asterism even after considerable bending.

It was observed that the Laue spots from the tension side appeared to be elongated in the direction approximately parallel to the specimen axis, whereas those on the compression side were elongated in the direction 90° to the former one (Fig. 7a,b). However, the asterism characteristic of the compression side was slightly curved thus indicating that crystallite rotation may have occurred about two or more axes. The asterism from the tension side was generally a line type streak for the usual film to specimen distance of 3 cm. However, when the Laue reflection from the tension side is enlarged by increasing the film to specimen distance to 12 cm, a very complex type of asterism is revealed (Figure 7c). It is evident, however, that the present observations concerning the type and extent of asterism suggest that the tension and compression sides of a bent molybdenum single crystal may be deforming as two different crystals.

Evidence was presented by Chen and Maddin⁽⁷⁾ for molybdenum single crystals deformed in tension that asterism is crystallographic in nature and represents a true account of plasticity in the specimen. It appears possible, therefore, to obtain information on the slip elements by plotting stereographically the crystallite rotations from the end points of Laue spots showing asterism. This was done in Figure 8 for the tension side of those specimens which showed considerable asterism of the line type streak. The crystallite rotations on the compression side were not determined due to the curvature of the Laue spots. Two of the specimens, M-25 and M-59, indicated a possible rotation toward the $\{111\}$ direction, while specimen M-181 gave slight evidence for rotation toward $\{111\}$. It is interesting to note that the crystallite rotations as determined from the extent of asterism were the same as the lattice or specimen axis rotations for specimens M-25 and M-59. The lattice rotation on the tension

side of M-181 gave a slight indication of movement toward $[111]$ and the crystallite rotation was towards $[111]$. The pole of the observed slip traces on the tension side of M-181 (Figure 8) appeared to indicate the operation of the slip plane (101) , which contains both of these directions. The present results of stereographic analysis of asterism indicate that in the determination of slip elements, it appears of value to consider the alterations in the structure as shown by the formation of asterism.

Yen and Hibbard⁽¹⁾ have observed a splitting of the Laue reflections into discrete spots after bending in two of their ten single crystal specimens of aluminum. They attributed this split-up of Laue spots to crystallite fragmentation. A break-up of Laue spots into individual areas was also observed in a single crystal of molybdenum bent through an angle of 15° at 2600°C ⁽⁹⁾ which was attributed to polygonization. In the present studies, division of the Laue reflections into high intensity areas was observed in all of the crystals bent (Fig. 9). However, these high intensity regions were always connected by diffuse areas. Consequently, it seems reasonable to interpret this phenomenon in terms of crystallite fragmentation where the individual crystallites are connected by high strain regions which may account for the diffuseness existing between the fragments producing the high intensity reflections.

Metallographic Observations

Microscopic examination of both the tension and compression sides of M-89 were made at low and high magnifications. Figure 10 shows representative photomicrographs of the markings at different positions around the periphery on the tension and compression sides. No marked difference was observable in the microscopic appearance of the markings on the tension and compression sides. Two distinct types of markings can be noted — deformation bands and slip lines. Whenever the slip lines could be clearly observed in the specimens examined, they were always straight. The deformation band, however, became wavy as it was followed around the specimen. The deformation band is represented by Figure 11 a, the

deformation band can be seen to split into two bands. The appearance of the bands illustrated in Figures 10a,b,d,e, are somewhat similar to the "slip lines" observed in extended sodium, potassium⁽¹⁰⁾ and mercury single crystals⁽¹¹⁾ at low magnifications. Stereographic analyses of the lines on more than one surface of M-89 appeared to indicate that the lines correspond to the $(\bar{1}01)$ plane. This result is in agreement with the X-ray data for the rotation on the compression side of M-89 (Figure 3), i.e., toward the $(\bar{1}01)$ pole.

Stereographic analyses of the slip lines on the tension side of specimens M-184, M-181, M-83 gave the following results: The pole of the observed slip traces for M-184 was in the vicinity of $(\bar{1}01)$, i.e., within $5^\circ - 10^\circ$, as illustrated in Figure 5. The lattice rotation on the tension side of this same crystal M-184, gave a definite movement toward the $[1\bar{1}1]$ direction which is contained in the $(\bar{1}01)$ plane. The slip plane on the tension side of M-181, as determined from slip traces, was near the (101) pole (Figure 6). The (101) plane might account for the lattice or crystallite rotations on the tension side of M-181. Although the pole of the slip traces for the tension side of M-83 was not located at any plane of low indices, it was closer to the (112) or (213) planes than to any of the $\{110\}$ planes (Figure 6). This is in agreement with previous observations of the extension of molybdenum single crystals with this initial orientation.⁽⁷⁾

It can be concluded from the present data that in three of the four cases where slip lines could be clearly observed, the pole of the slip traces was located near the $\{110\}$ planes.

Lattice-Deflection Data

Typical lattice deflection curves for molybdenum crystals under bending are shown in Figures 11 and 12. These curves indicate a normal type stress-strain

behavior with relaxation during loading. This relaxation occurred during the time when the X-ray patterns were obtained, with the load applied but not increasing for intervals of approximately one hour. It is believed that the load-relaxation shown here may be characteristic of the specimen rather than due to relaxation of the spring, since the spring was rated at 50 pounds and the loads were never above three pounds when these relaxations were observed.* The various load-deflection curves did not appear to vary significantly for different crystal orientations.

The maximum normal stress S_n for round specimens is given by the simple beam formula:

$$S_n = \frac{2PL}{3\pi r^3} \longrightarrow (1),$$

where P is the load in grams, L is the lever arm in mm (115 mm in the present work), and r is the radius of the specimen. The maximum normal stress for the onset of plastic deformation was of the order of $7\frac{1}{2}$ kg/mm² for the various crystals tested. Since no definite yield points were observed, the value of P was arbitrarily taken as the intersection of the best straight lines through the curve where the load-deflection initially showed a steep rise and the curve where the load-deflection is gradually flattened. If it is assumed that slip takes place under the same conditions as uniaxial loading, the resolved shear stress, S_s , along the operative slip plane may be calculated by multiplying equation (1) by $\sin\chi\cos\lambda$, where χ is the angle between the specimen axis and the major axis of the glide ellipse and λ is the angle between the specimen axis and the slip direction. For those specimens where the results appeared

* This belief is also supported by bending tests on 1/8 in. dia. steel rods in which no relaxation effect was observed using the same bending apparatus with loads up to 5 pounds and times up to 15 hours.

to indicate that the plane acting was $(101) [1\bar{1}1]$, the resolved shear stress in bending is calculated to be approximately $3\frac{1}{2} \text{ kg/mm}^2$ (5000 p.s.i.). Kochendorfer⁽³⁾ has reported that the critical shear stress observed in bending may be 1.7 times greater than that in uniaxial loading for round specimens. This might indicate a resolved shear stress of the order of 2 kg/mm^2 for molybdenum single crystals subjected to uniaxial loading. However, the values of resolved shear stress reported here can only be regarded as approximate because of the arbitrary value of P in equation (1) and because of the assumptions made in the calculations.

SUMMARY

A summary of the slip elements determined in this investigation is shown in Table I.

1. Lattice rotation during bending of molybdenum single crystals was found to occur toward the $\{110\}$ pole on the compression side and toward the $\langle 111 \rangle$ direction on the tension side.
2. Crystallite rotation determined from asterism and observations of slip traces appeared to indicate the participation of the $\{110\}$ planes and the $\langle 111 \rangle$ directions.
3. The results in (1) and (2) above support the suggestions of Chen and Maddin that the slip planes are of the type $\{110\}$ and the slip direction $\langle 111 \rangle$ for molybdenum single crystals deformed at room temperature.
4. The orientation change on the compression and tension sides of a molybdenum single crystal during bending are similar to that of two separate crystals deforming under compression and tension loading respectively.

5. A relaxation in load up to 0.5 pounds was obtained in the bending of molybdenum single crystals.
6. Laue reflections generally revealed a break-up which is interpreted as crystallite fragmentation.

ACKNOWLEDGMENTS

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TABLE I

SLIP NUMBER	SLIP ELEMENTS	
	<u>Compression Side</u>	<u>Tension Side</u>
11-35	$(\bar{1}01)^1$	—*
11-33	$(\bar{1}02)^1$	$\sim (112)$ or $(211)^3$
11-35	—*	$[111]^{1,2}$
11-23	$(\bar{1}01)^1$ and 3	$(\bar{1}01)^3$
11-27	$(\bar{1}01)^1$	$[111]^{1,2}$
11-50	$\sim (102)^1$	—*
11-131	$(\bar{1}01)^1$	$[\bar{1}11]^2, [\bar{1}11]^1, (101)^3$
11-104	—*	$[111]^1, (\bar{1}01)^3$

1. lattice rotation

2. extinction analysis

3. slip traces

* insufficient rotation for positive identification

TABLE I

<u>SAMPLE NUMBER</u>	<u>SLIP ELEMENTS</u>	
	<u>Compression Side</u>	<u>Tension Side</u>
M-35	$(\bar{1}01)^1$	—*
M-33	$(\bar{1}01)^1$	$\sim (112) \text{ or } (213)^3$
M-35	—*	$[111]^{1,2}$
M-38	$(\bar{1}01)^1 \text{ and } 3$	$(\bar{1}01)^3$
M-49	$(\bar{1}01)^1$	$[111]^{1,2}$
M-50	$\sim (101)^1$	—*
M-131	$(101)^1$	$[111],^2 [\bar{1}11],^1 (101)^2$
M-134	—*	$[111]^1 (101)^2$

1. lattice rotation

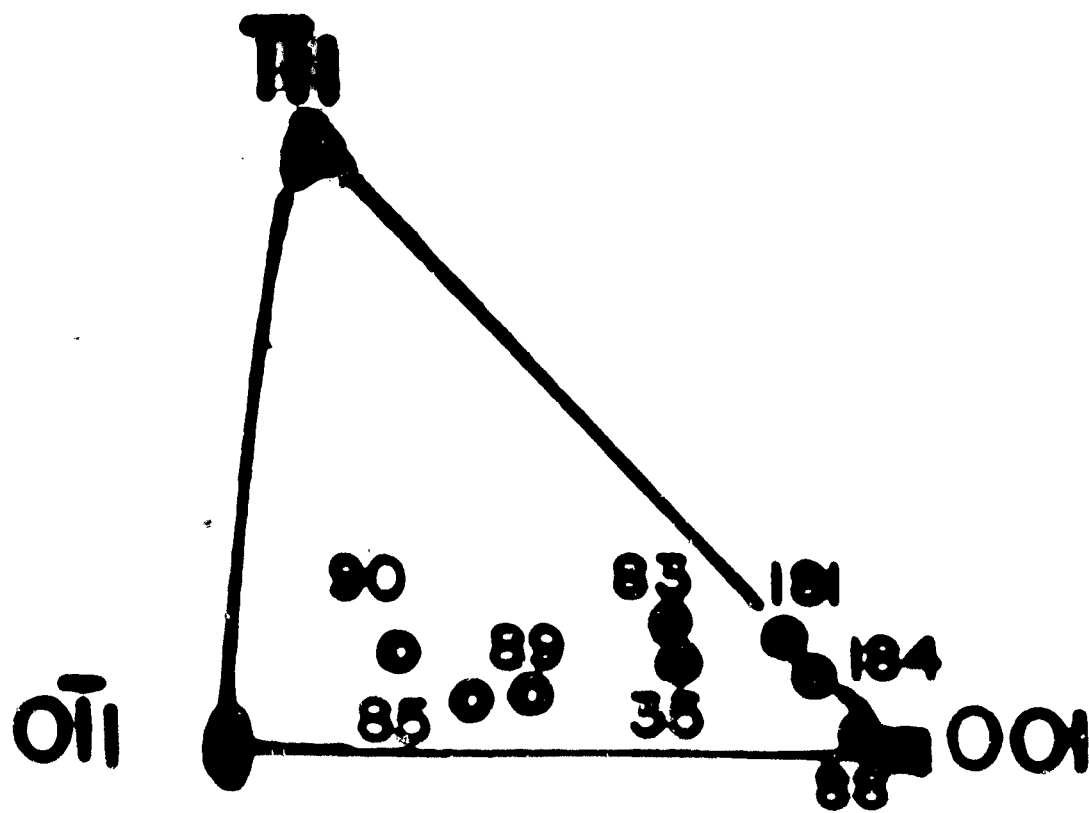
2. asterisk analysis

3. slip traces

* insufficient rotation for positive indication

FIGURES

- Figure 1. Bending apparatus assembled at X-ray machine; (a,b) Stationary bearings; (c) Single crystal specimen; (d) Movable steel plate; (e) Load cell; (f) Deflection cell.
- Figure 2. Orientations of crystals investigated.
- Figure 3. Projection of M-35, M-88, M-89 showing specimen axis rotations on the compression side.
- Figure 4. Projection of M-83, M-90, M-181 showing specimen axis rotations on the compression side.
- Figure 5. Projection of M-85, M-89, M-184 showing specimen axis rotation on the tension side and pole of observed slip traces for M-184.
- Figure 6. Laue photographs of compression side of M-181 (left) and tension side of M-85 (right) after increasing amounts of deflection from 0° to 16° and 12° respectively.
- Figure 7. Laue photographs of M-35 after bending through 13° . (a) tension side, (b) compression side (note change in direction of asterism); film to specimen distance 3 cm. (c) Same Laue reflections as in (a), but with film to specimen distance equal to 12 cm.
- Figure 8. Projection of M-¹⁸¹83, M-85, M-89 showing crystallite rotations obtained from asterism on the tension side and pole of observed slip traces for M-83 and M-181.
- Figure 9. Laue photographs of tension side of M-184 after increasing amounts of deflection from 0° to 15° showing a break-up of the Laue reflections into high intensity regions connected by diffuse areas.
- Figure 10. Deformation bands and slip lines of tension side (a)(b)(c) and compression side (d)(e)(f) of specimen M-89. Stress axis is horizontal. x200
- Figure 11. Load-deflection curves of specimens M-35, M-83, and M-88.



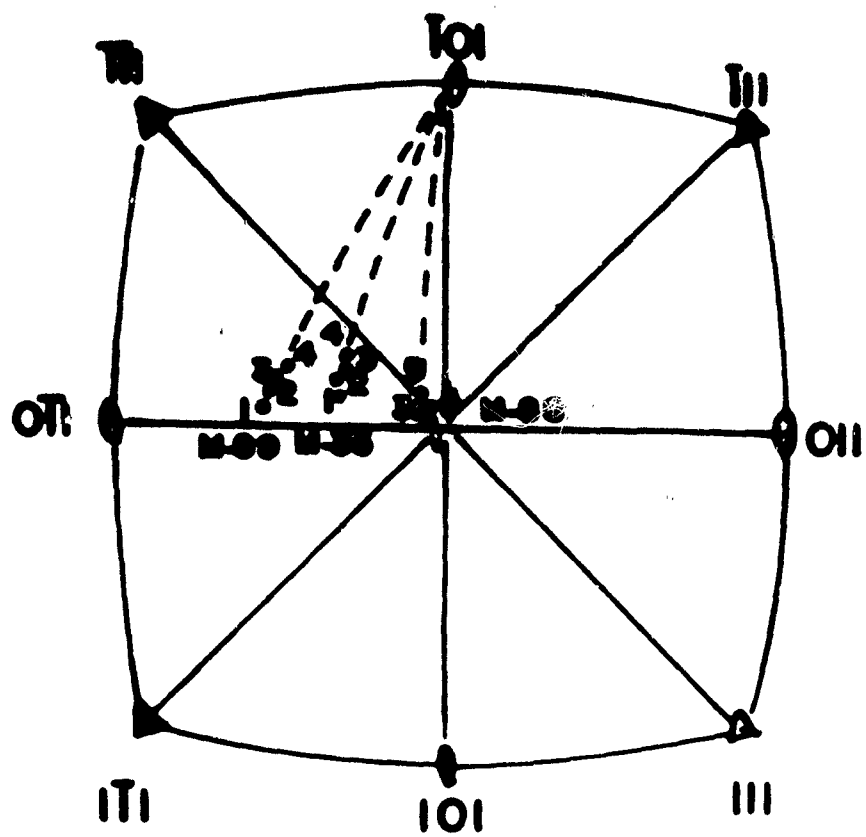
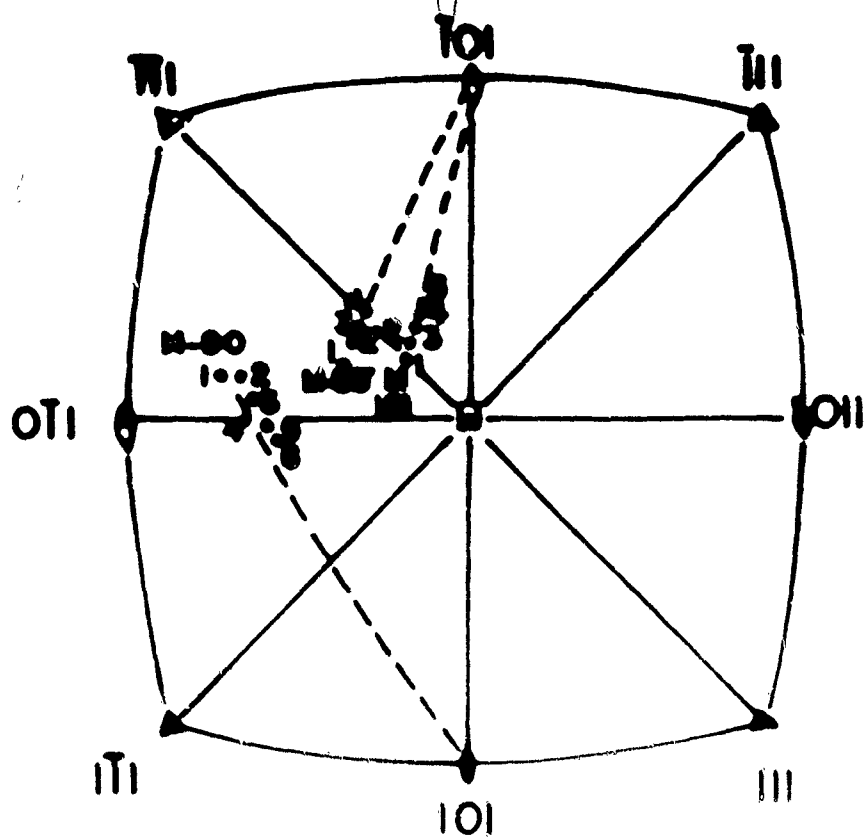


Fig 3



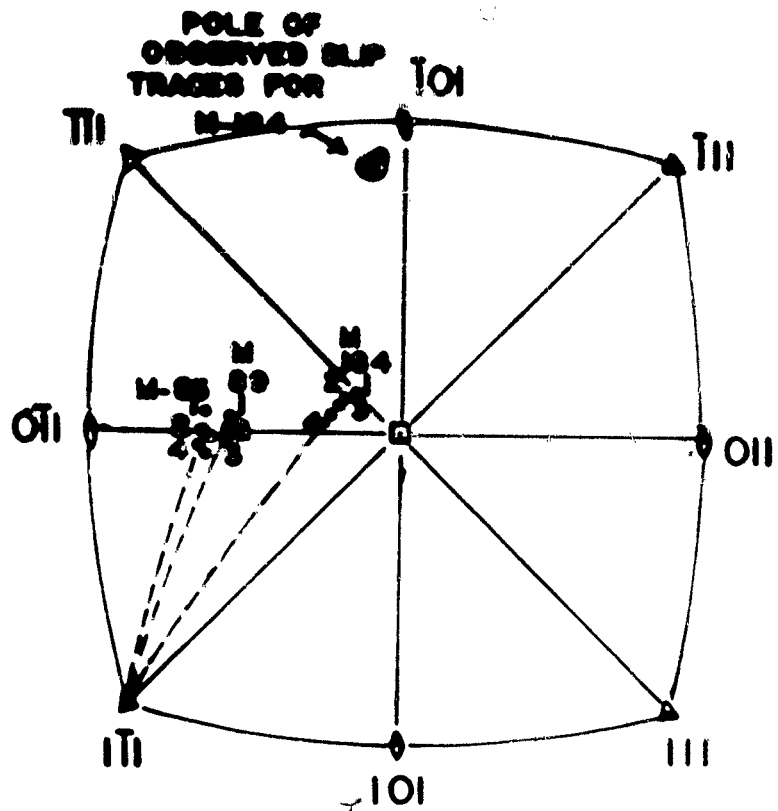


Fig 5

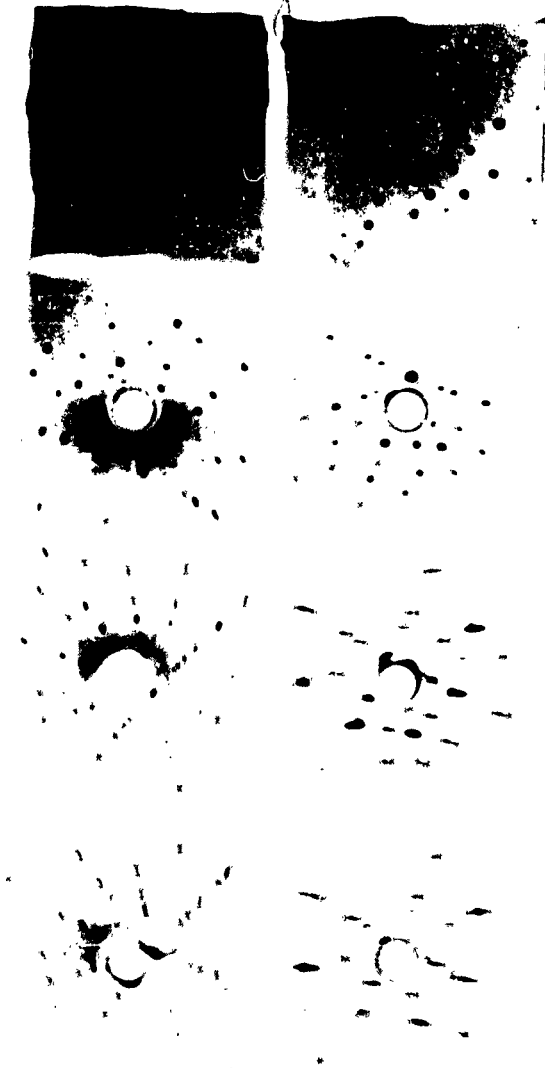




Fig 9

